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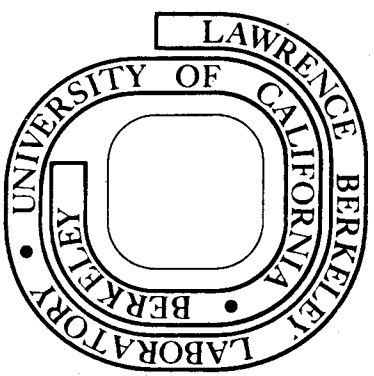

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CHANGES OF PREFERRED ORIENTATION IN HOT-PRESSED ALUMINA DURING CREEP

Tadaaki Sugita and Joseph A. Pask*

According to Hamano, Kinoshita and Oishi,[1] hot pressing of alumina results in preferred orientation of the alumina grains so that their basal planes tend to lie perpendicular to the pressing direction. The observed preferred orientation was hypothesized to be due to alignment or to preferential grain growth of the particles during hot pressing. Creep experiments were done to determine whether rearrangement of the grains during high temperature deformation could have contributed to the observed preferred orientation.

Alumina specimens were prepared by hot pressing Linde Al_2O_3 powder of 99.9% purity doped with 0.025 wt% of MgO in graphite dies at 1500°C for 60 min to 99.5% density. The average grain size was 3μm. Specimens, 5x5x10 mm, were cut with the long dimension perpendicular or parallel to the hot-pressing direction. The surfaces were ground smooth on a 400 grit diamond abrasive wheel. Creep tests were performed in uniaxial compression at 1450°C in air. The high temperature compressive deformation equipment was of the cantilever type described by Hulse and Copley.[2] Compressive strain was determined by measuring the movement of the top of the upper alumina rod (50 mm dia.) using a linear variable differential

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transformer in combination with a recording potentiometer. The apparatus was calibrated by measuring the movement of the alumina rod at the test temperature with no specimen under several loads. Orientations of grains were determined by examination of fracture surfaces with an X-ray diffractometer.

The incremental stress technique was used in the creep experiment. A stress was maintained constant until steady-state creep was reached and was then instantaneously increased or decreased and maintained constant until the creep rate was again steady.[3]

Figure 1 shows the creep results for specimens with the indicated relationships of the loading and hot-pressing directions. It is evident that the compressive creep deformation was accelerated when the creep direction was perpendicular to the hot-pressing direction (curve (a)). The stress exponents (slopes of the curves) are 1.8 and 1.6, respectively. This data is consistent with a grain boundary sliding model proposed by Langdon[4] and observed experimentally by Sugita and Pask.[5]

The labeled surfaces in Fig. 1 were examined by X-ray diffraction before and after creep. As pointed out by Hamano et al.[1] the ratio of the intensities of the Bragg reflections for the (014) and (113) planes from a given surface can be used as a measure of the alignment of the Al_2O_3 grains. A greater increase of the intensity of the (014) peak relative to a smaller increase or decrease of the (113) peak, and thus an increase of their ratio, indicates an increase of the preferred orientation of the grains with the basal planes parallel to the surface. A decrease of this ratio also indicates an increase of the alignment of the grains but with the basal planes now becoming more perpendicular to

the examined surface. As examples, Figs. 2 and 3 show the pertinent portions of the X-ray diffraction patterns for surfaces "3" and "1", respectively, of specimens oriented as in Fig. 1a. Table I lists the intensity ratios that were obtained from the indicated surfaces before and after creep at 1450°C for $\sim$15 h under stresses of 100 to 400 kg/cm^2. Similar data were obtained using integrated intensities.

A comparison of the ratios in Table I from surfaces before creep indicates that some preferred orientation occurred on hot pressing, as reported,[1] with the basal planes tending to be parallel to surface "3" which is perpendicular to the hot-pressing direction. After creep, the basal planes tended to align parallel to the surface perpendicular to the loading direction. Thus, for specimens whose creep direction was perpendicular to the hot-pressing direction (Fig. 1a), surface "1" on the basis of the change in ratios showed an increase in the degree of parallel alignment of the basal planes, surface "3" showed a decrease in parallel alignment, and surface "2" showed an increase in the alignment of the basal planes but perpendicular to the surface. The ratios for specimens whose creep direction was parallel to the hot-pressing direction (Fig. 1b) suggest an enhancement of the alignment of the basal planes perpendicular to this direction. The accelerated creep for the specimen with the hot-pressing direction perpendicular to the compressive creep direction shown in Fig. 1 is consistent with the observed realignment mechanisms.

Although the data is not rigorously quantitative, the trends support the hypothesis that grain rotations accommodated by grain boundary sliding[5] contribute to the preferred orientation occurring during compressive deformation and compaction.

Acknowledgments

The writers are grateful to M. Furukawa for providing the materials and in obtaining the X-ray diffraction patterns, to N. Tarumasu for the experimental work, and to Robert H. Bragg, K. Shyoji and M. Kinoshita for many discussions.

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References

1. Y. Hamano, M. Kinoshita and Y. Oishi, "Enhanced Grain Growth and Orientation in Hot-pressed Alumina," J. Ceram. Assoc. Japan, 70 [6] 165-71 (1962).

2. C. O. Hulse and S. M. Copley, "High Temperature Compressive Deformation Equipment," Bull. Am. Ceram. Soc., 45 [5] 530-4 (1966).

3. D. R. Cropper and T. G. Langdon, "Creep of Polycrystalline Lithium Fluoride," Phil..Mag., 18 [156] 1181-92 (1968).

4. T. G. Langdon, "Grain Boundary Sliding as a Deformation Mechanism During Creep," Phil. Mag., 22 [178] 689-700 (1970).

5. T. Sugita and J. A. Pask, "Creep of Doped Polycrystalline Al_2O_3," J. Am. Ceram. Soc., 53 [11] 609-13 (1970).

FIGURE CAPTIONS

Fig. 1. Effect of creep direction relative to hot pressing direction on creep rate of alumina at 1450°C.

Fig. 2. X-ray diffractometer patterns of hot-pressed alumina on surface "3" in orientation of Fig. 1a: (a) before creep, and (b) after creep at 1450°C for $\tilde{\sim}$15 h under stresses of 100 to 400 kg/cm^2.

Fig. 3. X-ray diffractometer patterns of hot-pressed alumina on surface "1" in orientation of Fig. 1a: (a) before creep, and (b) after creep at 1450°C for $\tilde{\sim}$15 h under stresses of 100 to 400 kg/cm^2.

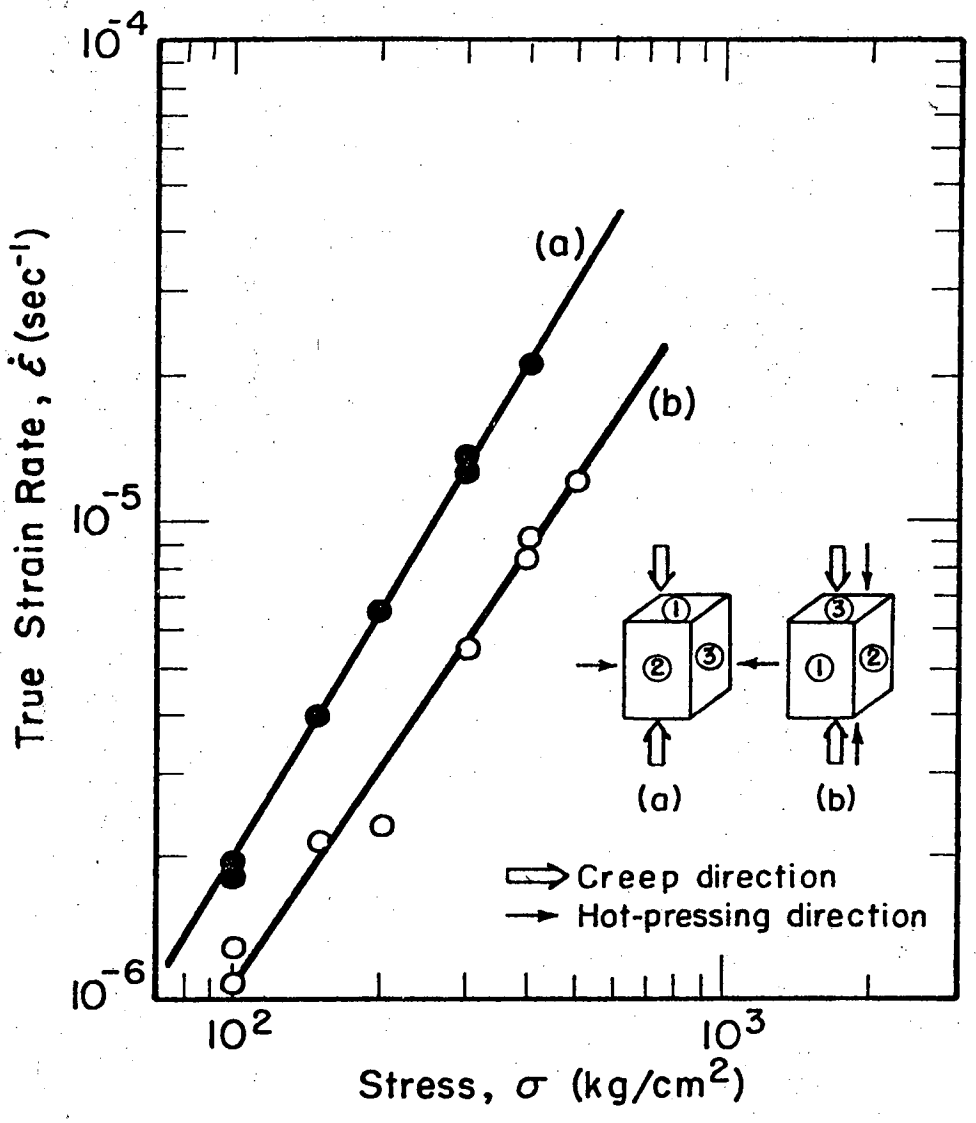

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Fig. 1

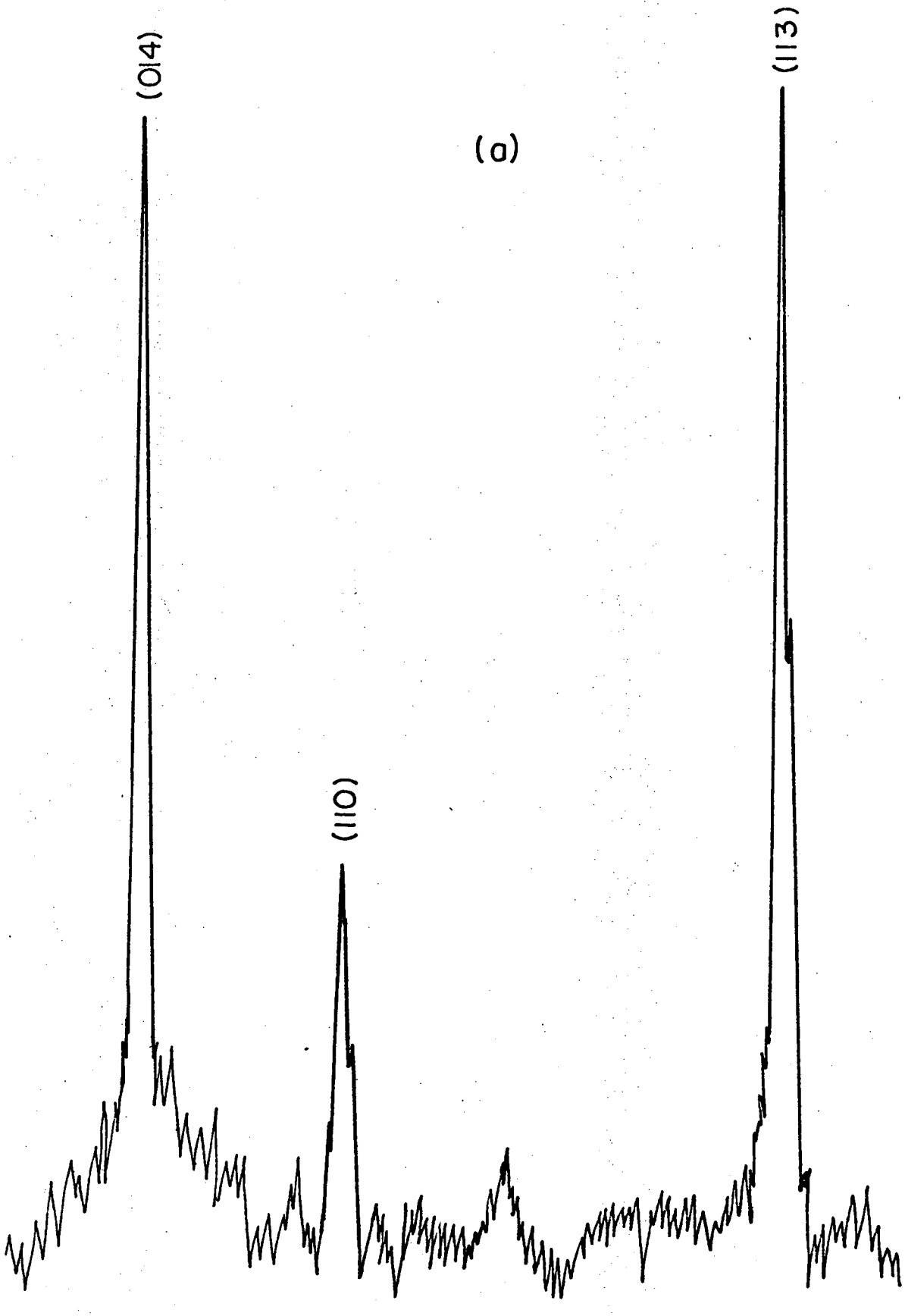

Fig. 2a

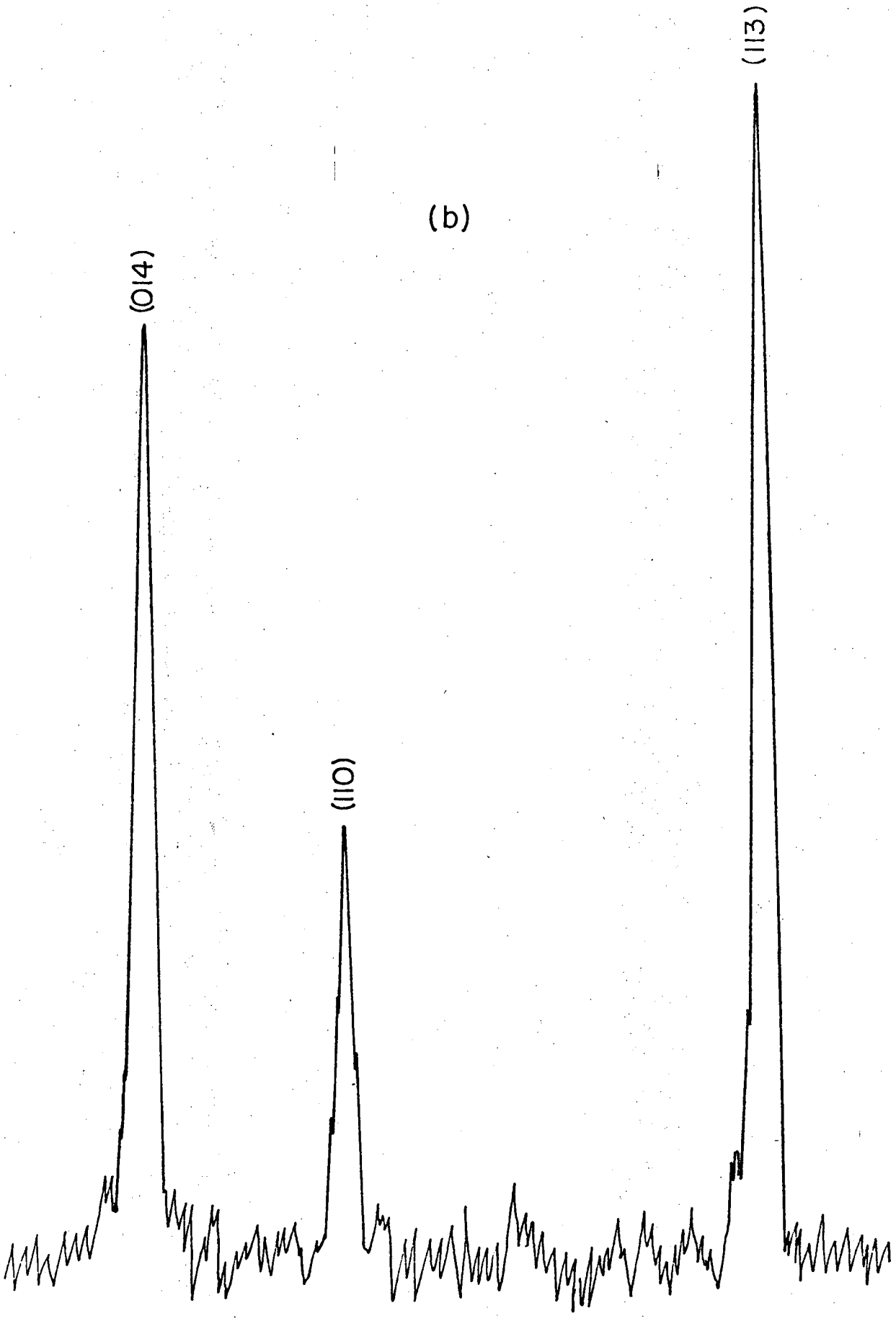

Fig. 2b

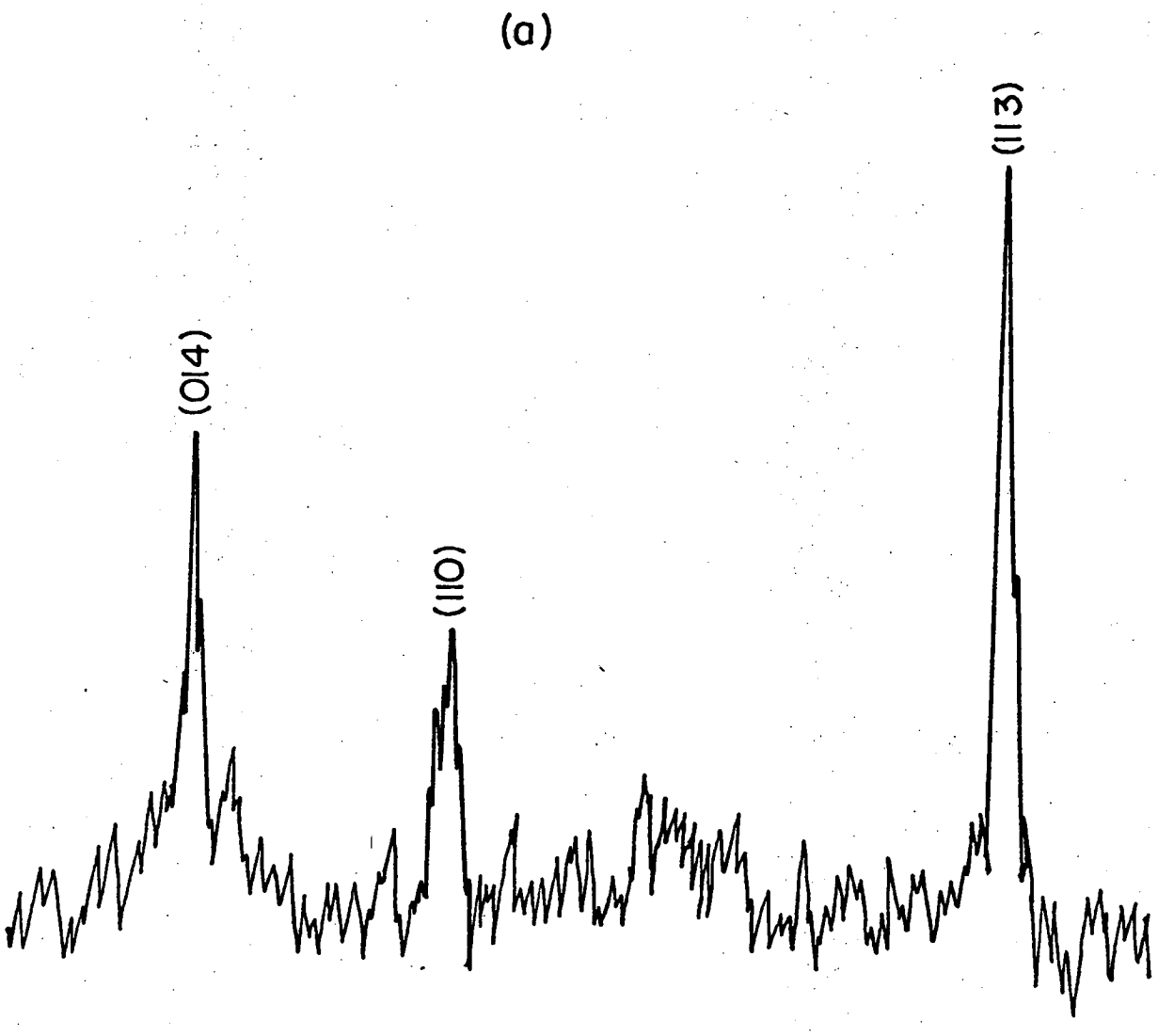

Fig. 3a

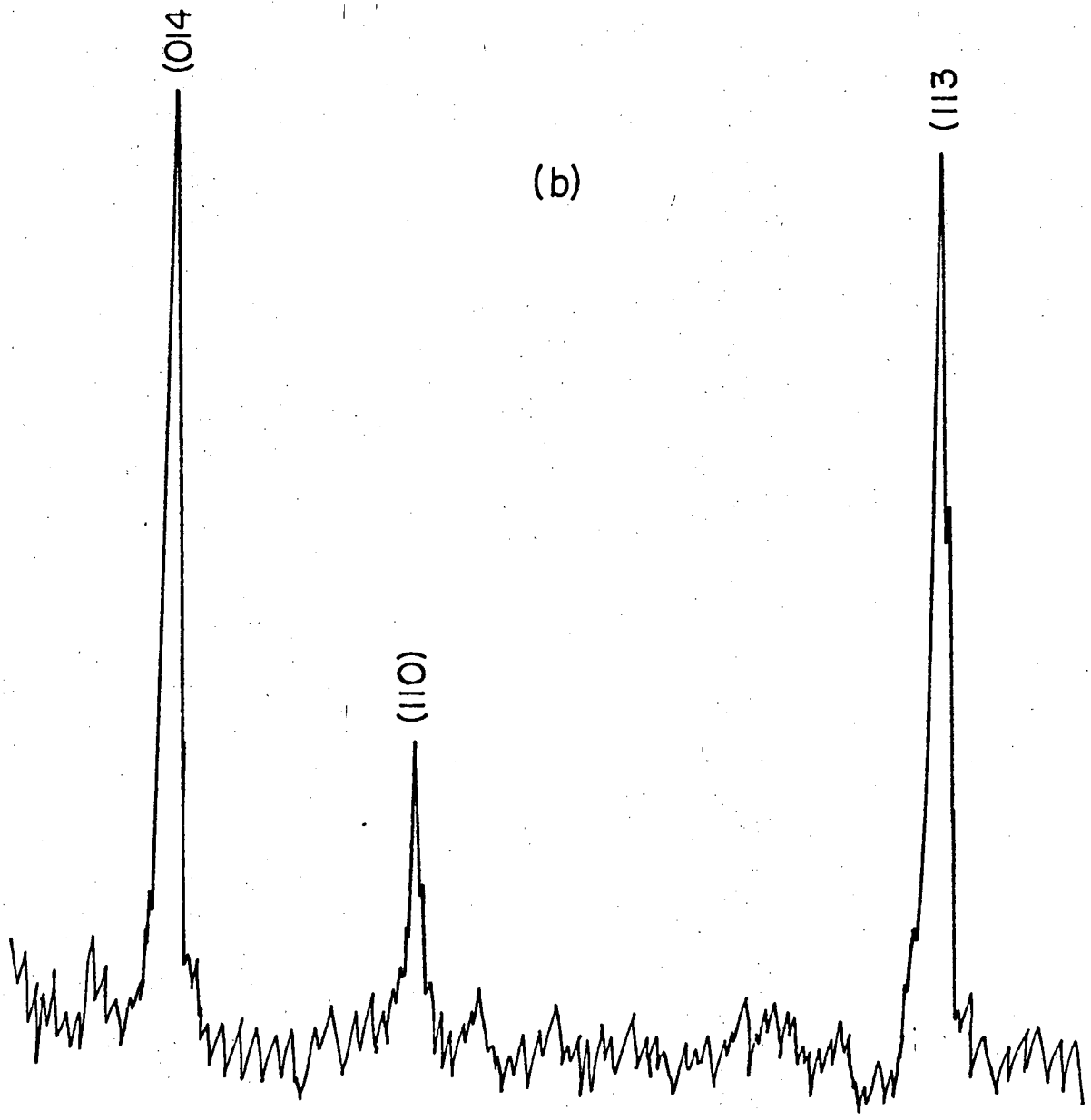

Fig. 3b

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